CENTRAL INTELLIGENCE AGENCY

25X1A

# INFORMATION REPORT

[redacted]

COUNTRY USSR

SUBJECT Comment on Technical Papers Dealing with Oxidation of Paraffin Wax

PLACE ACQUIRED (BY SOURCE) [redacted]

DATE ACQUIRED (BY SOURCE) 25X1A

DATE (OF INFO.)

OCT NO.

DATE DISTR. 21 DEC 53

NO. OF PAGES 5

NO. OF ENCLS.

SUPP. TO REPORT NO.



25X1X

THIS IS **UNEVALUATED** INFORMATION

[redacted]

COMPOSITION OF FATTY ACIDS DERIVED FROM OXIDIZED PARAFFIN - A G Drabkin and Z V Solovelchik - The Journal of Applied Chemistry of the USSR (Vol 23, 1950)

1. This article reports on a study concerning the composition of fatty acids derived from oxidized paraffin, wherein a crude oxidized paraffin having an acid number of approximately 86.5 and containing a large quantity of unsaponifiable material, hydroxy acids and ester derivatives was processed in a manner so as to recover essentially the straight-chained carboxylic acids from the complex mixture. Efforts were then made to characterize these fatty acids by molecular weight and type. The general method of purification of the crude acids involved extraction with petroleum naphtha or gasoline to remove a precipitate of the materials which were rich in hydroxy acids. In addition a zinc chloride solution was used to further precipitate hydroxy type materials, leaving in solution the so-called carboxylic acids. These acids from the naphtha solution were then saponified with caustic and treated so as to remove the unsaponifiable matter. The acids after having been freed of unsaponifiable matter were then converted to the methyl esters and these esters were subjected to fractional distillation.

2. The fractionated esters were then saponified with alcoholic alkali over a water bath and then the alcohol was driven off and the soaps were decomposed with hydrochloric acid. The resultant acids were extracted with ether. The ether extracts were washed with water and desiccated. The acids recovered after the solvent had been driven off were subjected to further investigation which involved determination of boiling range and the acid number. Carbon and hydrogen analysis were made on these acid fractions, and an effort was made to compare the recovered materials with the known structures of the common carboxylic acids.

SEE LAST PAGE FOR SUBJECT & AREA CODES

5

25X1A



[redacted]

3. It was concluded that the carboxylic acids recovered from this oxidized paraffin fraction were monobasic and possessed a normal structure. Acids were isolated from the fractions ranging in molecular size from about C-7 to C-23.

4. Regarding the significance of this work I feel that it should be pointed out that all of the procedures mentioned in this particular paper are well known to those familiar with the oxidation of paraffin waxes and the refining of the crude oxidates. They do not represent new or novel methods of refining or separation of the acids. Further the methods and the techniques used in this work have been reported previously in both the German and US literature in some detail and also in some of the older Soviet literature.

5. As one example of the publication of a very similar type of work at an earlier date, reference is made to Fantzen, et al. Fette U Seifen, 45, 388 (1938). This report covers the composition of the fatty acids from the oxidation product from Fischer-Gatsch in which the analysis of the hydrocarbon is given, followed by an explanation of the processing which also involved the formation of the methyl ester and the fractionation of the methyl esters. The composition of the fatty acids derived therefrom are in relative agreement with that presented in this paper and the products ranging from about $C_7$, $H_{14}$, $O_2$ to fractions having molecular weights higher than $C_{18}H_{33}O_2$. There are other investigators who do not agree that the acids present in oxidized paraffin are as simple in structure as Drabkin and Soloveichik conclude.

6. The work described in this paper is of the nature that requires considerable amount of relatively tedious laboratory investigation, however, it does not represent any significant advance in the knowledge of the composition of wax acids or improvement in the processing of acids from oxidized waxes.

THE OXIDATION OF PARAFFIN WAX DISTILLATE - A G Drabkin and Z V Soloveichik - Journal of Applied Chemistry of the USSR (Vol 23, 1950)

7. The work reported in this paper covers an investigation of the oxidation characteristics of a mixture of solid paraffin and mineral oil. This fraction would correspond in US refinery terminology to a crude wax distillate. The material used in this work consisted of some 30% solid paraffin with the remainder of the material being oil. In the introduction to this paper it is indicated in a brief sentence that the USSR has an interest in the preparation of consistent greases using the oxidized waxes as a fat substitute. It further points out that paraffin wax was the most suitable starting material developed to date and that this work was involved in attempting to utilize the oxidized oily waxes which would, of course, be in greater supply and lower in cost than the oxidized refined paraffin wax. The actual oxidation studies described do not represent any novel oxidation methods or procedures. They are those which are commonly used in the oxidation of petroleum fractions and the results are somewhat as would be expected with the role of the mineral oil being about what has been ascribed to it in numerous other publications.

8. In general, the oily waxes containing high percentages of oil oxidize with more difficulty and at a slower rate than do the refined paraffin waxes. These oily waxes also result in dark colored products high in hydroxy acid content and high in naphtha insoluble acid contents. The findings in this paper do not differ from the previously indicated properties of the oxidates from oily waxes. It is pointed out that by purification procedures on the distillate that increased oxidation rates can be obtained. This has been well known for some time. The Germans have pointed out that acid treating with sulfuric acid or the treatment with aluminum chloride increased the susceptibility of the oily waxes to oxidation. This particular paper does not give any data on the use of these oxidized oily

waxes or oxidized wax distillates for use in grease making. However, in checking into this matter further I have located a reference which indicates that the Soviets are actively engaged in attempting to prepare satisfactory greases from the soaps of the oxidized paraffin waxes. The following is a reference to one such effort on their part. Reference: Oxidized Paraffin As a Raw Material for the Production of Calcium Soap Greases by V K Tsyskoskie. Azerbaidzhanskoe Neftyanoe Khoz, 26 #8, 17-21 (1947). This paper indicated that a water stabilized calcium grease has been prepared from an oxidized paraffin wax. Their tests on this grease indicated it to be, in their opinion, roughly equivalent to water stabilized calcium soap greases prepared from natural fats. However, in reviewing the tests on these greases it was evident that a larger amount of water of stabilization was used in each case with the petroleum oxidate type greases in the range of three to four per cent whereas something in the range of one to two per cent would normally be used with the conventional fatty material. Further the soap content or the amount of oxidized paraffin wax required (26.5%) was approximately twice that of the natural fat which would have been normally used to make a grease of equivalent consistency. From the performance tests given in the paper it was not possible to ascertain the exact quality of the greases which had been prepared from the oxidized fractions, however, claims were made that these greases did exhibit satisfactory performance characteristic in plant and automotive equipment.

9. It would appear that even though the properties of the grease described here may not be fully equivalent to a conventional grease prepared from a natural fat, the Soviets may be capable of making usable greases from the oxidized petroleum fractions. In the event that the economics within the Soviet Union permitted such an action it apparently would be within their capability to utilize oxidized paraffin wax as a source of fatty material for use in greases. It would not be expected that the substitution of the oxidized wax fractions could be made in all greases particularly some of the specialty greases and it would not necessarily follow that the quality of the greases from the oxidized wax would be fully equivalent in all respects to the quality obtained with the wide variety of natural fats which are used in grease making. However, from our knowledge of greases from oxidized petroleum fractions, it would be expected that the Soviets would be capable of making greases which would be usable in a relatively large number of applications.

10. The article by Drabkin and Soloveichik does not contain any particularly outstanding disclosures. It would be interesting to obtain additional information on the extent and degree to which the Soviets are conducting work on the utilization of oxidized fractions for grease making, as this could conceivably represent a large saving in their natural fats which in turn would increase fat availability for edible materials and other critical applications.

THE HIGH TEMPERATURE OXIDIZATION OF PARAFFIN WAX - A K Plise and A I Bykovets from the Organic Chemistry Laboratory of the Odessa Technological Institute of the Food and Refrigeration Industry - The Journal of Applied Chemistry of the USSR (Vol 24, 1951)

11. This paper reports on what appears to be relatively preliminary experiments on the high temperature oxidation of paraffin wax with the primary objective being the reduction in oxidation time required without a degradation in quality of the oxidate.

12. A great deal of the early work on the oxidation of paraffin that was conducted prior to 1920 was concerned with high temperature oxidation, that is, temperatures above about 150°C. However, it has been known for sometime that high temperature oxidations of the normal type result in acids of a degraded quality primarily from the standpoint of excess amounts of hydroxy type acids that are formed and the dark colors of the products. These hydroxy acids are undesirable in the preparation of synthetic edible fats and they are also undesirable in some other applications such as in lubrication oil additives, etc, due to their mineral oil insolubility. Consequently, most of the successful commercial oxidation of paraffin wax has been conducted somewhere in the range of 220°F to possibly 250°F. The lower temperature oxidations favor high yields of carboxylic type acids and lower amounts of the hydroxy acids. The lower temperatures also produce lighter colored products, which can be more easily refined and purified to an edible form of fat or for use in other applications. It is true that the low temperature oxidations will require longer periods of oxidation. Consequently, larger reaction vessels would be required. An advantage would be expected if the reaction could be conducted at the higher temperatures while maintaining high quality of products, as the reaction time would be shorter and the thruput or capacity of a given plant would be considerably increased. This would in effect, result in a reduction in cost of the fatty material prepared by the oxidation of paraffin wax.

13. This particular paper covers some work on efforts to oxidize paraffin wax for very short periods of time in the order of one minute while using temperatures in the range of 200 to 500°C. The experimenters claim that the optimum temperature range is 300 to 400°C and report a relatively rapid oxidation at those temperatures showing neutralization number or acid number values in the range of 20 for the one minute of contact time. It is further claimed that the amount of hydroxy acids formed is essentially nil. The data in the table on page 1359 indicate the presence of no hydroxy acids, however, it also shows that the hydroxyl number is relatively high (140 to 150). Although the method of analysis for hydroxy acids is not shown there would be some question as to whether or not their analysis is correct if they have a high hydroxyl number yet claim the practical absence of hydroxy acids.

14. This type of oxidation, that is using the relatively short contact time at an increased temperature, should lend itself to some form of a continuous process.

15. Although not mentioned in the particular paper, most of the oxidation procedures which have been in commercial use in Germany and in other locations, have been batch processes. In general, there would be certain economic advantages if the oxidation procedure could be conducted on a continuous basis. The German literature reports a number of efforts to develop a continuous process, however, the continuous processes have usually resulted in a degradation of product quality.

16. The fact that this work was apparently done at the Odessa Technological Institute of the Food and Refrigeration Industry may indicate that the Soviet Union is concerned in this particular work with the problem of preparing edible fats from the oxidation of paraffin. This would be a continuation or further work on the type of process that was operated in Germany during World War II. The fact that considerable emphasis is placed on the absence of hydroxy acids which would be very undesirable in any type of edible fatty material, further indicates that these investigators were interested in the utilization of the oxidates for edible fats. Since the preparation of the edible fats type of oxidized material was conducted in Germany at relatively low temperatures in the order of 100°C, in order to obtain a light colored product with as few undesirable side reactions as possible and with as low a yield of hydroxy acids as possible, it appears that what the investigators here may have in mind is the operation of the process at a higher temperature with a much shorter contact time and thereby reducing the cost of the preparation of the fatty materials. The work covered by this paper is considered to be relatively preliminary in nature in that very little information on

[redacted]

the nature of the oxidates is given with regard to their physical and chemical properties. Further, it is to be expected that at the low neutralization numbers reported on in this paper, that is in the order of 2 to 20 or 30, that only a small amount of hydroxy acids would be formed in the low temperature process also. A commercial operation involving oxidation to only 20 neutralization number would not normally be attractive due to the small amount of paraffin converted to acids. However, if this oxidation were continued and, in fact, re-cycled through this apparatus, so that contact time was increased sufficiently to raise the neutralization number to a value of, for example, 80 to 100, it would be expected that the amount of hydroxy acids would increase even at low temperatures oxidations. In using the high temperatures involved here, there is a possibility that the rate at which the hydroxy acids increase with regard to neutralization number might be greater than at the lower temperatures. It is interesting to note here, however, that the Soviet Union apparently is continuing to work with some vigor on various phases of oxidation work and attempting to improve the existing oxidation procedures as well as developing various utilizations for the oxidates.

25X1A

- end -

LIBRARY SUBJECT & AREA CODES

668.393 N
668.664 N
614.7 N
614.87 N
614.641 N